REMARKS

Applicant's attorney is appreciative of the interview granted by the Examiner on September 22, 2003. At that interview, the terminology to which objection has been raised was discussed, and it was agreed that a new substitute specification would be submitted.

Objection has been raised to the abstract on conflicting grounds, first that the application does not contain an abstract (page 2 of Office Action) and second, that the application contains two abstracts (page 5 of office action).

An abstract was added to the application in the paper filed on August 13, 2002, and the originally filed abstract was canceled in the paper filed on March 12, 2003. Thus a single abstract is in the application, and Applicant submits that this abstract is clearly written.

Regarding the drawings, new Figures 1 and 2 have been submitted. In these figures:

Fig. 1 has been labeled "Prior Art;"

"M2" in Fig. 1 has been removed;

"10" in Fig. 2 has been removed; and

"V" in Fig. 2 has been removed.

Moreover, in Example 1 of the specification, "100" now designates "underflow," and in Example 3, "An" has been changed to "1n."

Based on the above changes, Applicant believes that all objections to the drawings have been removed.

Objection has been raised to the amendment of August 13, 2002 as containing new matter under 35 USC 132. The substitute specification filed on August 13, 2002 has been withdrawn, and a new substitute specification is filed herewith. In the new specification, the objected to terminology has been withdrawn, except for the designation of

solid content as "g/ 1 aluminate." As explained at the interview, this designation is identical to the original French text, and describes the solid content in the slurry. The original translation as g aluminate/ liter was an incorrect translation.

In addition, it is noted the designation "CPFT" for the amount of solid smaller than a given size has been removed in the new substitute specification.

The other changes in the substitute specification are minor in nature, and the new substitute specification does not contain new matter.

Claims 7-14 have been rejected under 35 USC 112, 1st paragraph, as containing subject matter not adequately described in the specification.

The claims have now been extensively amended to remove terminology to which objection has been raised, and to clarify the process steps. In this regard, "CPFT" has been removed from the claims, and has been replaced by "percent [or particles] finer than..." in accordance with the explanation given in the specification on page 3. It is not known whether there are still objections to claim 7, but it is noted that the process of determining the relationship R is discussed in detail in the originally filed specification at page 8, lines 11-31, and the discussion of trigger thresholds is found on page 9, lines 9-23 of the originally filed specification.

The limitations of claims 8, 9, 11, 13 and 14 have been objected to as being new matter. In this regard, it is noted that claim 8 corresponds to claim 2 as filed and claim 9 corresponds to claim 3 as filed. Claim 11 is supported by Example 1 at the top of page 14 of the originally filed specification.

Claim 13 is a recitation of steps a1)-a4) on pages 7-8 of

the specification, and claim 14 is a recitation of steps b1)-b4) on page 10 of the specification.

All claims have been amended to the extent possible to use terminology found in the specification as originally filed.

Withdrawal of this rejection is requested.

A further rejection of claims 7-14 under 35 USC 112, 1st paragraph, has also been made.

A question has been raised as to how the relationship R is determined in claim 7, with the comment that it does not appear to be based on CPFT X1 and CPFT X2, in view of the specification, page 7, that it is based on $\%<X2(t)$ and $\%<X1(t-t)$. However, these disclosures are actually the same, since CPFT X1 is the same as $\%<X1$, and CPFT X2 is the same as $\%<X2$. Claim 7 has been amended to remove "CPFT" and recites instead that the "percent smaller than" is measured over time (t) to determine R. Thus, there is no conflict.

Moreover, the recitations regarding "controlling the circuit" and "updating the correlation" (now "forming a correlation) are explained in detail in the explanation of Example 2 on page 16 of the specification.

Withdrawal of this rejection is requested.

Claims 7-14 have been rejected under 35 USC 112, 2nd paragraph, as indefinite.

In claim 7, "predetermined" has been alleged to be indefinite based on the *Seagram* case from 1950. As discussed at the interview, Applicants do not believe that this case is an accurate reflection of the current state of patent law and practice, and should not be cited.

In claim 7, the terms "regularly" and "updating" have been deleted.

As to the "corrective action," claim 7 has been amended

to clarify that this step brings the amount finer than X2 within the maximum permissible variation previously defined in the claim. This process is explained in the specification on page 6.

The corrective action itself may be adjustment of the solid content of the slurry, or other actions explained on page 6, lines 16-24.

Claim 9 has been amended to recite modifying amounts of liquor added to the first feed tank and first agglomeration tank, as explained at page 6, lines 25-29. Thus, the amount fed to each tank may be varied to achieve a desired result.

In claims 13 and 14, the "feed tank series" has been changed to "circuit" to provide antecedent basis.
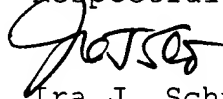
The other objection to claim 13 is not understood, since, as explained above, there is no conflict between "CPFT" and "%<X."

As to the concept of "accidental phenomenon," this is explained in the specification on page 8, lines 2-4. Thus, an event may be observed on both of the determined curves, separated by a period of time, and this separation is used to determine a relationship between the two curves.

Withdrawal of this rejection is requested.

In view of the foregoing amendments and remarks, Applicant submits that the present application is now in condition for allowance. An early allowance of the application with amended claims is earnestly solicited.

Respectfully submitted,

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PROCESS FOR CONTROLLING THE PARTICLE SIZE IN A BAYER CIRCUIT PRECIPITATION SYSTEM, INCLUDING AN AGGLOMERATION PHASE

~~Technical domain~~ Field of the invention

The invention relates to the precipitation of alumina trihydrate according to the Bayer process, carried out in an American type precipitation system including a preliminary agglomeration phase.

5

~~State of prior art~~ Description of related art

The Bayer process can produce alumina from bauxite ore, particularly alumina designed to be transformed

10 into aluminum by igneous electrolysis. According to this process, the bauxite ore is treated when hot by means of an aqueous sodium hydroxide solution with an appropriate concentration in order to obtain a suspension containing pregnant sodium aluminate liquor

15 and insoluble residues. After separation of these residues, the pregnant sodium aluminate liquor, also called Bayer liquor, is decomposed by seeding with recycled aluminum trihydroxide particles until aluminum trihydroxide grains (also called alumina trihydrate or

20 hydrargillite) are obtained, and which are themselves then calcinated to obtain an alumina with particular particle size distribution and physicochemical properties. The sodium aluminate liquor depleted in alumina (spent liquor) is then recycled to digest the

25 ore, possibly after being concentrated, by the evaporation and addition of sodium hydroxide or caustic soda.

The productivity of the liquor during its crystallisation is defined by the quantity of alumina restored in the form of alumina trihydrate by crystallisation of the pregnant liquor, related to a given volume of pregnant liquor. The productivity expressed in kilograms of alumina per cubic meter of liquor (kg of Al_2O_3/m^3), is related to the caustic concentration in the pregnant liquor. In general, this concentration in American type Bayer processes is close to 100-130 g of Na2O/ liter, which is lower than in European type Bayer processes, and this explains why a productivity in the crystallisation of the pregnant liquor is considered to be good when it exceeds 70 kg of Al_2O_3/m^3 for an American type Bayer process, or when it exceeds 80 kg of Al_2O_3/m^3 for a European type Bayer process.

The difference between European type and American type Bayer processes is in the solid content of the slurry during precipitation. The slurry is the result of introducing a recycled alumina trihydrate seed into the aluminate liquor, and part of the alumina in solution changing to the solid phase. We will define the solid content in the slurry as the weight of solid particles present in the slurry per unit volume of pregnant aluminate liquor entering into the precipitation workshop (and not per unit volume of the suspension).

Alumina to be transformed into aluminum by igneous electrolysis must have a number of properties, including:

- o good ~~pourability~~ flowability so that electrolysis tanks can be continuously supplied with controlled quantities of alumina,

o a high dissolution rate,

o a low tendency to dusting.

These properties are closely related to the morphology and the particle size distribution of

5   alumina grains, themselves closely related to the morphology and particle size distribution of hydrargillite particles formed during the precipitation. It is particularly important to limit the proportion of very fine particles that can be

10   classified in two main categories: fines (for which the average diameter is between 10 and 50 µm) and ultrafines, for which the average diameter is less than 10 µm. Since a good correlation is observed between the particle size distribution of alumina and the

15   particle size distribution of the production hydrate from which it is derived, an attempt is made to control the particle size of the ~~rotating~~ hydrate circulating ~~used~~ in the precipitation series, and particularly in the crystal growth phase. Thus for example, in order

20   to obtain a good quality "metallurgical" alumina, an attempt is made to obtain a ~~rotating~~ circulating hydrate for which the amount passing a 45 µm sieve is less than 10%, ~~in other words~~ i.e. a suspension containing less than 10% of particles with a diameter

25   of less than 45 µm. In the rest of this discussion, we will denote the quantity passing X micrometers as "%<X".

Concerning the American type Bayer process, the precipitation comprises a preliminary agglomeration

30   phase characterized by a particularly low solid content. In patent US 4 234 559, the precipitation circuit comprises firstly a series of agglomeration tanks and then a series of feed tanks and finally three

classification tanks (primary, secondary, tertiary).
While the hydrate produced is derived from the
underflow from the primary classification tank, the
fines seed originating from the underflow from the

5 tertiary classification tank is inserted in controlled
quantities into the series of agglomeration tanks and
the larger seed originating from the underflow from the
secondary classification tank is added into the series
of feed tanks. Since the fines are destroyed during

10 the agglomeration phase, the problem of controlling the
particle size of the hydrate produced does not arise.

But attempts are ~~always~~ still being made to
increase the productivity of the American type Bayer
process, which is lower than the productivity of

15 European type Bayer cycles. In US 5 158 577 and
EP 0 515 407, only part of the pregnant liquor is added
into the agglomeration ~~system~~ tank series, and the rest
is added directly to the crystal growth. This can ~~give~~
result in different solid contents in these two parts

20 of the crystallization system, a low solids content ~~of~~
~~dry material~~ in agglomeration tanks which is essential
if it is required that agglomeration should take place
under good conditions, and a high solid content in feed
tanks which can increase productivity.

25 But an instability in the precipitation is observed
if the solid content ~~of dry material~~ is increased at
the feed and if the number of agglomeration tanks is
limited, with a serious risk of the sudden appearance
of large quantities of fines in the ~~rotating~~

30 circulating hydrate (particle size crisis). This type
of crisis should be avoided, since if ~~it is not~~
~~corrected~~ no correction is made, the particle size

quality of the produced hydrate is strongly deteriorated.

The particle size instability is due to a reduction in the ratio between the sum of the production <u>hydrate</u> and the fine seed sent to agglomeration, and the ~~rotating~~ <u>circulating</u> hydrate. A reduction in this ratio makes it impossible to implement effective corrective actions when a drift of the amount passing 45 μm (% < 45) is observed on the ~~rotating~~ <u>circulating</u> hydrate.

~~Statement of the problem~~ Summary of the invention

Therefore, the applicant attempted to define a process that could increase the productivity of the American type Bayer process by preventing unacceptable particle size fluctuations, particularly the sudden appearance of large quantities of fines and ultrafines in the ~~rotating~~ <u>circulating</u> hydrate.

~~Purpose of the invention~~

The process developed by the applicant is a process for controlling the precipitation of an American type Bayer circuit including a preliminary agglomeration phase, a crystal growth phase and a classification phase, in which the particle size quality of the hydrate produced is monitored by measuring the amount of rotating hydrate passing X2 μm in feed tanks, characterized in that it comprises:

    a) a preparation step carried out once and for all, intended firstly to setup a relation R in intensity and in time between ~~rotating~~

circulating hydrate material passing X1 µm and material passing X2 µm, where X1 is less than X2, and secondly to define trigger thresholds on the value of material passing X1 µm, starting
5 from the maximum authorized variation interval on values passing X2 µm;

b) control of the process itself, carried out during the installation operating period which, apart from the daily measurement of material
10 passing X2 µm and a regular update of the correlation between the said material passing X2 µm and the particle size of the hydrate produced, a daily measurement of the ~~rotating~~ circulating hydrate passing X1 µm and a regular
15 update of the relation R between the said material passing X1 µm and the said material passing X2 µm, and triggering of corrective action in the slurry at the beginning of the precipitation when the measured value of
20 material passing X1 µm reaches one of the regularly updated trigger thresholds determined in the previous step.

This corrective action in the slurry at the beginning of precipitation may be a modification to the
25 temperature of the aluminate liquor added into the agglomeration tank, the addition of additives at the beginning of the crystal growth system such as "Crystal Growth Modifiers" described in US 4 737 352, recycling a part of the end of ~~crystallisation~~ crystallization
30 slurry, or preferably, modification of the solid content in the slurry in the first agglomeration tank.

The solid content in the slurry in the agglomeration phase may be modified simply by adding

more or less pregnant aluminate liquor in the first agglomeration tank, the remaining aliquot being directed to the feed tank. If the quantity of material passing X1 µm is too great, there are too many fines; the amount of pregnant aluminate liquor fed into the agglomeration tank must be increased. If the amount passing X1 µm is too low, there is a risk that productivity will drop; the pregnant aluminate liquor feed from the agglomeration tank ~~needs to~~ must be reduced.

The preliminary step, carried out once only, is intended to determine the relation R and trigger thresholds for the values of X1 µm that will be used at the beginning of application of the process control. This preliminary step comprises the following steps:

a1) Daily measurement of material passing X1 µm in the slurry at a particular point in the precipitation system, which is used to produce a first particle size time diagram represented by a curve $Y = \% < X1(t)$.

a2) Daily measurement of material passing X2 µm in the slurry at a particular point in the precipitation system, which is used to produce a second particle size time diagram represented by a curve $Y = \% < X2(t)$ and in which X2, greater than X1, is a value already known for its good correlation with the particle size of the hydrate produced. For example, it may be material passing 45 µm measured in slurry at pump-off.

a3) Creation of an empirical relation between the two particle size time diagrams, the purpose of which is to characterize the relation between

the variation of the population of material passing X1 µm and the variation of the population of material passing X2 µm, in intensity and in time. This relation R may be written in the form:

$$R(\% < X2(t), \% < X1(t-\tau)) = 0$$

where t is the date on which material passing X2 µm is measured and τ is a characteristic time interval estimated by observing the occurrence of the same accidental phenomenon on each curve (the same type of extreme, an inflection, etc.).

a4) Definition of the maximum threshold and the minimum threshold of material passing X1 µm, obtained from the relation R previously established and a maximum interval of the authorized variation of values of material passing X2 µm.

The purpose of the relation R is to predict the variation in the particle size, in other words to anticipate crises, by observing the variation in the population of the finest hydrate particles (~~with~~ which have a size of less than X1 µm). The applicant observed that material passing X1 µm ~~anticipates~~ enables anticipating a change in the material passing a higher X2 value; an accident on the time diagram $\% < X1(t)$ is amplified and shifted in time on the $\% < X2(t)$ time diagram. The time shift "τ" is higher as the difference between X1 and X2 increases. In practice, a value X2 greater than 40 µm (normally 45 µm) will be chosen, and the value of X1 will be taken to be less than or equal to 20 µm.

The measurement point is preferably at pump-off, but it may take place earlier, provided that it remains within the crystal growth series. Measurement points for material passing X1 and for material passing X2 may
5   be different. However, they must remain the same throughout the process control and must be as far as possible from the points at which disturbing additions are made irregularly in the slurry.

Measurements referred to as being "daily" are
10 regular measurements and although they are not necessarily daily, they are sufficient frequent to give useable time diagrams.

Concerning the required variation interval of material passing X2 μm, a maximum is defined above
15 which it is known that the particle size of the hydrate produced is no longer satisfactory (too many fines) and a minimum is defined below which it is known that economic operating conditions become bad.

The trigger thresholds are thus determined from the
20 time diagram for the material passing X1 μm, taking account not only of the maximum authorized variation interval on the values of material passing X2 μm, but also the uncertainty of the measurement of material passing X1 μm and the stability of the efficiency of
25 the hydrate classification system.

This preparatory step, at the end of which the time diagrams, the relation R and the trigger threshold become operational, is of the order of three months. But this step can be accelerated either by deliberately
30 triggering an excess creation of fines, or by analyzing previous particle size results if they contain the required information about material passing X1 and X2 μm.

Since precipitation is a complex phenomenon that depends on a large number of parameters (particularly the composition of the treated bauxite that may change in time), instead of using a relation R established once and for all, it is better to use a relation that is regularly updated. Similarly, the correlation between the value of material passing X2 μm and the particle size of the hydrate produced must be updated regularly. This regular updating may be applied at a less frequent rate than daily measurements (for example monthly).

The actual process control comprises the following phases:

b1) A daily measurement of the material passing X1 μm in the slurry at a particular point in the precipitation system, in order to complete the first particle size time diagram represented by the curve $Y = \% < X1(t)$.

b2) A daily measurement of the material passing X2 μm in the slurry at a particular point in the precipitation system, in order to complete the first particle size time diagram represented by the curve $Y = \% < X2(t)$.

b3) Regular updating(for example monthly) of the empirical relation R between the two particle size time diagrams and the definition of trigger thresholds of material passing X1 μm, or updating after an important modification in a process parameter.

b4) Triggering of a corrective action in the slurry at the beginning of the precipitation when the measured value of material passing X1 μm reaches one of the thresholds defined in b3).

This corrective action is preferably a modification to the solid content in the slurry in the first agglomeration tank. If ~~it is a~~ the maximum threshold is reached, there are ~~two~~ too many fines, ~~the~~ and the solid content ~~of dry material~~ is reduced by introducing a stronger aliquot of the pregnant aluminate liquor into the first agglomeration tank. If ~~it is a~~ the minimum threshold is reached, the feed of pregnant aluminate liquor into the agglomeration tank is reduced and the ~~crystal growth~~ feed into feed tanks is increased.

As for the preparatory step, empirical relations are determined starting from daily particle size measurements made on the ~~rotating~~ circulating hydrate. As for the preparatory step, the daily rate is not necessarily daily, but is sufficiently frequent to be able to produce useable time diagrams. Trigger thresholds on the "X1 µm passing material" curve are also deduced from the particle size time diagram; they take account of the uncertainty of the measurement of material passing X1, the maximum authorized variation interval on values of material passing X2 µm, and the stability of the efficiency of the hydrate classification system.

For relatively low solid content ($\approx$ 350 g/l aluminate ~~of aluminate/ liter~~), it is sufficient to measure amount passing X1 = 20 µm. This may be done using instruments that diffract laser beams and make mass determinations (Malvern Mastersizer, Cilas, etc.).

But in order to increase productivity, much higher solid content must be achieved, comparable to those achieved in European type processes, in other words greater than 700 g/l aluminate ~~of aluminate/ liter~~.

However, as the solid content in feed tanks increases, the need for a low value of X1 also increases. When the solid content is high, a longer time is necessary to correct particle size disturbances; therefore, it
5 is necessary to have a greater time shift τ; and this time shift is proportional to the difference between X1 and X2.

In this case, it is preferable to measure the amount passing 10 µm, or even a lower value (down to
10 1.5 µm). When X1 becomes this low, it becomes necessary to use a celloscopic measurement (COULTER or ELZONE) that determines counts rather than mass. These measurements are more difficult, but the economic consequences are negligible.

15 Productivity can also be increased by increasing the caustic content of the aluminate liquor. The applicant has observed that this method of controlling the particle size can be applied very well on industrial installations with concentrations reaching
20 160 g of Na_2O/ liter.

Concerning the corrective action, it is also recommended that an empirical relation should be set up in advance to quantify the effects of the said action, for example during the preparatory phase. Thus, in
25 order to quantify the effects of the modification to the solid content in the first agglomeration tank, the first step will be to establish the actual relation between the said solid content and the proportion of destroyed fines.

30 **Brief description of the drawings**

Fig. 1 is a schematic diagram of a prior art Bayer circuit; and

Fig. 2 is a schematic diagram of a Bayer circuit according to the invention.

Embodiments of the invention - Examples

5   The embodiment of the invention will be better understood from the following description of a number of examples.

Figures 1 and 2 show the part of the Bayer circuit corresponding to the precipitation phase, the
10   precipitation being of the American type with a preliminary agglomeration step.

Figure 1 illustrates the first example typical of prior art.

Figure 2 illustrates subsequent examples describing
15   two embodiments of this invention.

These embodiments are described in examples 2 and 3. To simplify the presentation, we have illustrated the classification phase ~~will~~ with all conventional classification devices in example 1. In fact, and
20   particularly with the increase in the solid content, ~~an expert in the subject~~ one of ordinary skill in the art could select additional or replacement systems such as cyclones or filters.

25   ### Example 1 (Prior art)

The pregnant aluminate liquor **1** enters the precipitation circuit at a temperature of about 75°C, enters the first agglomeration tank **A**. It has a caustic concentration of 130 g of Na2O/ liter.
30   The tertiary seed **9**, composed essentially of fine and ultrafine particles, is added into the first agglomeration tank **A** and is mixed with pregnant liquor. The resulting slurry **2** passes through a sequence of

agglomeration tanks such that practically all fines and ultrafines have disappeared after an average residence time of about 5 hours. During the agglomeration phase, the solid content in the slurry has increased from
5 20 g/l aluminate ~~of aluminate / liter~~ to about 30 g/l aluminate ~~of aluminate / liter~~.

At the exit from the agglomeration series, the slurry **3** is added into the first feed tank **N** with the secondary seed **8**, and the addition increases the solid
10 content by 80 g/l aluminate ~~of aluminate / liter~~. The new slurry **4** passes through a sequence of feed tanks with an average residence time of about 20 hours, and cooling by about 5°C. At the exit from the feed, the slurry **5**, called the "pump-off", ~~contains~~ has a solid
15 content of about 175 g/l aluminate ~~of dry aluminate material / liter~~. By measuring the amount of this slurry **5** passing 45 µm at the exit from the crystal growth **M1**, the particle size quality of the production hydrate can be estimated so that an optimum variation
20 interval can be defined for this material passing 45 µm. Under the conditions in the example, approximately 10% of material passing 45 µm will give a good quality "metallurgical" alumina.

The slurry **5** at the exit from the crystal growth is
25 then added into the first classifier tank **PT**. The ~~overflow~~ underflow **100** from the first classifier **PT** produces production hydrate and the overflow **6** that has a solid content of 100 g/ l aluminate ~~of aluminate / liter~~, is added into a second classifier tank **ST**. The
30 underflow from the second classifier tank **ST** acts as a secondary seed **8** that is reinjected at the beginning of the crystal growth **N** and the overflow **7**, which then has

a solid content of only 20 g/ liter, is sent to a third classifier tank **TT**. The overflow **10** from the third classifier tank is the spent aluminum liquor that is reinjected as the bauxite green liquor at the beginning

5 of the Bayer cycle, after concentration by evaporation and the addition of caustic soda. The overflow **9** from the third classifier tank is added back as the tertiary seed into the first agglomeration tank **A** after filtration and washing.

10

Example 2: Precipitation according to the invention with an average solid content in the ~~rotating~~ circulating hydrate

15   The pregnant aluminate liquor **1** that enters the precipitation circuit at a temperature of about 75°C, is separated into two aliquots, the first aliquot **1a** representing about one third of the total liquor being added into the first agglomeration tank **A**, and the

20 second aliquot **1n** being added into the first feed tank **N**. The caustic concentration in the pregnant aluminate liquor **1** is 130 g of ~~Na2O~~ Na_2O/ liter.

The tertiary seed **9a**, composed essentially of fine and ultrafine particles, is added into a first

25 agglomeration tank **A** after filtration and partial or complete washing, and is mixed with the pregnant liquor. The resulting suspension **2** follows a sequence of agglomerating tanks such that a predefined proportion of fines and ultrafines has disappeared

30 after an average residence time of 5 hours. During the agglomeration phase, the quantity of hydrate is

increased by 10 g/l aluminate ~~of aluminate / liter~~ entering the workshop (1a and 1n).

The slurry **3** at the exit from the agglomeration series is added into the first feed tank **N** with the

5 second aliquot **1n** of pregnant aluminate liquor, the rest of the ~~washed or unwashed~~ tertiary seed **9n**, washed or unwashed, and the secondary seed **8**, the addition of which increases the solid content by 220 g/l aluminate ~~of aluminate / liter~~. The new slurry **4** follows a

10 series of feed tanks with an average residence time of 20 hours and cooling of 5°C. At the exit from the crystal growth, the slurry **5** ~~contains~~ has a solid content of about 350 g/l alumniate ~~of dry aluminate material / liter~~.

15 The slurry **5** at the exit from the crystal growth is added into a first classifier tank **PT**. The underflow **100** from the first classifier **PT** supplies the production hydrate, and the overflow **6** which has a solid content of 250 g/l aluminate ~~of aluminate / liter~~

20 is added into a second classifier tank **ST**. The underflow from the second classifier tank **ST** acts as a secondary seed **8** that is reinjected at the beginning of the crystal growth **N** and the overflow **7**, which then only has a solid content of 30 g/ liter, is sent to the

25 third classifier tank **TT**.

A measurement **M1** of the material passing 45 µm, and a measurement **M2** of the material passing 20 µm, are made in the slurry every day at pump-off, using a laser diffraction apparatus.

30 Observations of the particle size quality of the hydrate produced can be used to define the authorized variation interval on values of material passing 45 µm,

and the target set value C on the material passing 20 µm is defined, by means of the empirical relation defined during the preparation phase and continually updated afterwards.

5   Depending on the difference between M2 and the value of the set value C defined in advance to guarantee the particle size quality of the product and the differences M2 - C obtained in the previous days, the quantity of fines to be agglomerated in addition to 10 or less than the previous day, are determined.

During the preparation phase, the relation between the solid content in the agglomeration phase and the proportion of destroyed fines was determined. This relation is used to fix the aliquot **1a** used in the 15 agglomeration. This aliquot **1a** fixes the solid content in the agglomeration tanks and therefore the change required for destruction of the fines.

Furthermore, starting from measurements **M1** and **M2** made on previous days, the relation between the set 20 value C and the required level M1 for the material passing 45 µm, and secondly the relation between the solid content and fines destroyed in the agglomeration, are adjusted.

25 **Example 3**: **Precipitation according to the invention with a high solid content in the ~~rotating~~ circulating hydrate**

The pregnant aluminate liquor **1** arriving in the precipitation circuit at a temperature of about 75°C, 30 is separated in two aliquots, the first **1a** representing about half of the total liquor being added into the first agglomeration tank **A**, the second **1n** being added

into the first feed tank **N**. The caustic concentration
in the pregnant liquor **1a** is 130 g of ~~Na2O~~ Na_2O/ liter.

The filtered tertiary seed **9**, composed essentially
of fine and ultrafine particles, is added into the
agglomeration step **A** and mixed with the fraction 1a of
pregnant liquor. The resulting slurry **2** stays in the
agglomeration phase for about 5 hours. The proportion
between the quantity of tertiary seed and the flow of
liquor 1a is adjusted such that a predefined proportion
of fines and ultrafines disappears. The quantity of
hydrate during the agglomeration phase is increased by
15 g/l ~~of~~ aluminate entering in the workshop ~~/ liter~~
(**1a** and ~~An~~ **1n**).

At the exit from the agglomeration series, the
slurry **3** is added into the first feed tank **N** and with
the second aliquot **1n** of pregnant aluminate liquor and
the secondary seed **8**, the addition of which increases
the solid content by 840 g/l aluminate ~~of aluminate /~~
~~liter~~. The new slurry **4** passes through a series of
feed tanks with an average residence time of 18 hours
and cooling by **10°C**. At the exit from the crystal
growth, the slurry **5** ~~contains~~ has a solid content of
about 1000 g/l aluminate ~~of solid aluminate content/~~
~~liter~~.

At least one measurement **M1** of the material passing
45 µm is made every day at this location using a laser
diffraction apparatus, and a measurement **M2** of the
material passing 1.5 µm is made with an ELZONE counter.

The slurry **5** at the exit from the crystal growth is
added into a first classifier tank **PT**. The underflow
100 from the first classifier **PT** supplies the
production hydrate and the overflow **6**, that has a solid

aluminate content of 870 g/l aluminate ~~liter~~, is added into a second classifier tank **ST**. The underflow from the second classifier tank **ST** is used as a secondary seed **8** that is reinjected at the beginning of the

5 crystal growth **N** after filtration and the overflow **7**, that has a solid content that no longer exceeds 30 g/ liter, is sent to the third classifier tank **TT**.

The process for monitoring and controlling the particle size of the ~~rotating~~ hydrate in the slurry at

10 pump-off is identical to that described in example 2.

~~Advantages of the process according to the invention~~

The process according to the invention has the following advantages:

- Particle size fluctuations related to the increase in the inertia of the system originating from the increase in solid contents are avoided.
- The productivity of liquors can thus be increased:
    - by controlling crystallization at high solid contents without endangering the quality of the alumina produced,
    - by adjusting the pump-off particle size to the maximum level compatible with quality requirements and the production classification system.